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(19) (CA) APPLICATION FOR CANADIAN PATENT (12)

(54) Preparation of Polymers Based on 1-Vinylimidazoles

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Notice: This application is as filed and may therefore contain an incomplete specification.



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The preparation of polymers based on 1-vinylimidazoles

Abstract

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Polymers based on vinylimidazoles are prepared by free-radical precipitation polymerization in an organic solvent or solvent mixture which contains no aromatic groups and, apart from oxygen, no heteroatom.

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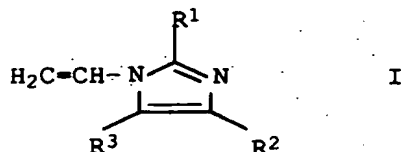
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We claim:

1. A process for preparing polymers based on vinylimidazoles by  
5 polymerizing a monomer mixture composed of

(a) 10-100% by weight of a compound of the general formula I

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where R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> are identical or different and each is hydrogen, C<sub>1</sub>-C<sub>18</sub>-alkyl or phenyl,

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(b) 0-90% by weight of another monoethylenically unsaturated monomer capable of free-radical copolymerization, and

(c) 0-20% by weight of a monomer which contains at least two non-conjugated ethylenic double bonds,

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in the presence of compounds which form free radicals, wherein the polymerization is carried out in an organic solvent or solvent mixture which contains no aromatic groups and, apart from oxygen, no heteroatom and in which the resulting polymer is of low solubility or insoluble.

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2. A process as claimed in claim 1, wherein the organic solvent or solvent mixture is selected from the group of saturated hydrocarbons with 5-12 carbon atoms, of dialkyl ethers with 2-12 carbon atoms, of C<sub>4</sub>-C<sub>16</sub>-alkanols, of C<sub>3</sub>-C<sub>12</sub>-ketones and  
35 of C<sub>1</sub>-C<sub>22</sub>-alkyl esters of C<sub>1</sub>-C<sub>22</sub>-carboxylic acids.

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3. A process as claimed in claim 1, wherein heptane, cyclohexane or ethyl acetate is used as organic solvent.

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The preparation of polymers based on 1-vinylimidazoles

The present invention relates to a novel process for preparing  
5 polymers based on 1-vinylimidazole by precipitation polymeriza-  
tion in the presence of compounds which form free radicals.

1-Vinylimidazoles are normally polymerized in aqueous or ethan-  
olic solution. To prepare higher molecular weight or crosslinked  
10 polymers or special copolymers it is frequently more favorable to  
use the precipitation polymerization method.

EP-A 162 388 discloses the preparation of 1-vinylimidazole  
copolymers by precipitation polymerization in benzene.

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Furthermore, A. Chapiro et al., Eur. Polym. J., 24 (1988), 1019  
describe the precipitation polymerization of 1-vinylimidazole in  
benzene, toluene or tetrachloromethane. The preparation of  
copolymers of 1-vinylimidazole and 4-aminostyrene by precipita-  
20 tion polymerization in benzene is described by R.F.C. Bay et al.,  
Polymer, 32 (1991), 2456.

The polyvinylimidazoles prepared in tetrachloromethane have, how-  
ever, only low molecular weights, while the polymers obtained in  
25 benzene or toluene result as crosslinked gels which are difficult  
to work up.

An additional disadvantage of this known process is that the  
solvents used are toxicologically very objectionable.

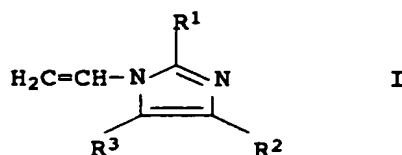
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It is an object of the present invention to find a process which  
allows the use of toxicologically less objectionable reaction  
media.

35 We have found that this object is achieved by a process

(a) 10-100% by weight of a compound of the general formula I

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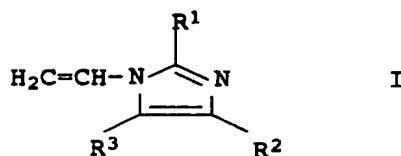
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where  $R^1$ ,  $R^2$  and  $R^3$  are identical or different and each is hydrogen,  $C_1$ - $C_{18}$ -alkyl or phenyl,

- (b) 0-90% by weight of another monoethylenically unsaturated monomer capable of free-radical copolymerization, and
- (c) 0-20% by weight of a monomer which contains at least two non-conjugated ethylenic double bonds,
- 10 in the presence of compounds which form free radicals, wherein the polymerization is carried out in an organic solvent or solvent mixture which contains no aromatic groups and, apart from oxygen, no heteroatom and in which the resulting polymer is of low solubility or insoluble.
- 15 Suitable monomers (a) are vinylimidazoles of the formula I

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- 25 where  $R^1$ ,  $R^2$  and  $R^3$  are identical or different and are each hydrogen,  $C_1$ - $C_{18}$ -alkyl or phenyl, for example 1-vinylimidazole, 2-methyl-1-vinylimidazole, 2-ethyl-1-vinylimidazole, 2-propyl-1-vinylimidazole, 2-butyl-1-vinylimidazole, 2,4-dimethyl-1-vinylimidazole, 2,5-dimethyl-1-vinylimidazole, 2-ethyl-4-methyl-1-vinylimidazole, 2-ethyl-5-methyl-1-vinylimidazole, 2,4,5-trimethyl-1-vinylimidazole, 4,5-diethyl-2-methyl-1-vinylimidazole, 4-methyl-1-vinylimidazole, 4-ethyl-1-vinylimidazole, 4,5-dimethyl-1-vinylimidazole, 5-methyl-1-vinylimidazole, 2,4,5-triethyl-1-vinylimidazole, 2-phenyl-1-vinylimidazole, 2-undecyl-1-vinylimidazole or 2-stearyl-1-vinylimidazole. It is also possible to use mixtures of said monomers in any desired ratios. Monomers of group (a) which are preferably used are 1-vinylimidazole, 2-methyl-1-vinylimidazole, 4(5)-methyl-1-vinylimidazole, 2-ethyl-1-vinylimidazole and 2-ethyl-4(5)-methyl-1-vinylimidazole. 1-Vinylimidazole and 2-methyl-1-vinylimidazole are very particularly preferred. The monomers are used in amounts of 10-100% by weight, preferably 25-100% by weight.

- Suitable monomers (b) are further monoethylenically unsaturated monomers capable of free-radical copolymerization, or mixtures thereof, for example N-vinyl lactams such as N-vinylpyrrolidone and N-vinylcaprolactam, N-vinylloxazolidinone, N-vinyltriazole,

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- N-vinyl-N-methylacetamide, (meth)acrylic esters such as methyl, ethyl, hydroxyethyl, propyl, hydroxypropyl, butyl, ethylhexyl, decyl, lauryl, i-bornyl, cetyl, palmityl, phenoxyethyl or stearyl acrylate or the corresponding methacrylates, (meth)acrylamides
- 5 such as acrylamide, N-methylolacrylamide, N,N-dimethylamino-propylacrylamide, N-tert-butylacrylamide, N-tert-octylacrylamide, N-undecylacrylamide or the corresponding methacrylamides, vinyl esters with 2-30, in particular 2-14, carbon atoms in the molecule such as vinyl acetate, vinyl propionate, vinyl laurate,
- 10 vinyl neooctanoate, vinyl neononanoate, vinyl neodecanoate, styrene, vinyltoluene,  $\alpha$ -methylstyrene, unsaturated carboxylic acids such as acrylic acid, methacrylic acid, crotonic acid, maleic acid, fumaric acid, itaconic acid or their anhydrides, 2-acrylamido-2-methylpropanesulfonic acid, acrylic esters having a basic
- 15 nitrogen atom such as diethylaminoethyl acrylate, dimethylaminoethyl acrylate, dimethylaminopropyl acrylate or the corresponding methacrylates, 2-vinylpyridine, 4-vinylpyridine. Particularly preferred are N-vinylpyrrolidone, N-vinylcaprolactam, alkyl (meth)acrylates, vinyl acetate, styrene, acrylic acid, meth-
- 20 acrylic acid, maleic acid and monomers which have a basic nitrogen atom. N-Vinylpyrrolidone is very particularly preferred. The monomers are used in amounts of 0-90% by weight, preferably 0-75% by weight.
- 25 Suitable monomers (c) are those compounds which are capable of free-radical copolymerization and which contain at least two non-conjugated ethylenic double bonds in the molecule. Examples of suitable monomers (c) are diacrylates or dimethacrylates or at least dihydric saturated alcohols, eg. ethylene glycol diacry-
- 30 late, ethylene glycol dimethacrylate, 1,2-propylene glycol diacrylate, 1,2-propylene glycol dimethacrylate, 1,4-butanediol diacrylate, 1,4-butanediol dimethacrylate, hexanediol diacrylate, hexanediol dimethacrylate, neopentyl glycol diacrylate, neopentyl glycol dimethacrylate, 3-methylpentanediol diacrylate and
- 35 3-methylpentanediol dimethacrylate. The acrylates and methacrylates of alcohols with more than 2 OH groups can also be used as monomers (c), eg. trimethylolpropane triacrylate or trimethylolpropane trimethacrylate. Also suitable are diacrylates or dimethacrylates of polyethylene glycols or polypropylene glycols
- 40 with molecular weights of, in each case, 100-9000. Polyethylene glycols and polypropylene glycols used to prepare the diacrylates or dimethacrylates preferably have a molecular weight of, in each case, 200-2000. Apart from the homopolymers of ethylene oxide and propylene oxide it is also possible to use block copolymers of
- 45 ethylene oxide and propylene oxide or copolymers of ethylene oxide and propylene oxide which contain the ethylene oxide and propylene oxide units in random distribution. The oligomers of

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ethylene oxide and propylene oxide are also suitable for preparing the crosslinkers, eg. diethylene glycol diacrylate, diethylene glycol dimethacrylate, triethylene glycol diacrylate, triethylene glycol dimethacrylate, tetraethylene glycol diacrylate and/or tetraethylene glycol dimethacrylate. Also suitable as crosslinkers are vinyl esters of ethylenically unsaturated C<sub>3</sub>-C<sub>6</sub>-carboxylic acids, eg. vinyl acetate, vinyl methacrylate or vinyl itaconate. Also suitable as crosslinkers are vinyl esters of saturated carboxylic acids containing at least 2 carboxyl groups, and di- and polyvinyl ethers of at least dihydric alcohols, eg. divinyl adipate, butanediol divinyl ether and trimethylolpropane trivinyl ether as well as acrylamides or methacrylamides of at least difunctional saturated amines such as methylenebis(acrylamide) or ethylenebis(methacrylamide). Further suitable monomers (c) are allyl esters of ethylenically unsaturated carboxylic acids, eg. allyl acrylate and allyl methacrylate, allyl ethers of polyhydric alcohols, eg. pentaerythritol triallyl ether, triallylsucrose and pentaallylsucrose. Also suitable as crosslinkers are methylenebismethacrylamide, divinylethyleneurea, divinylpropyleneurea, divinylbenzene, divinylidioxane, tetraallylsilane, tetravinylsilane, 1,7-octadiene, diallyl phthalate, trivinylcyclohexane, 1,9-decadiene or triallyltriazinetriene. Divinylethyleneurea, allyl methacrylate and diacrylates and dimethacrylates of at least dihydric alcohols are particularly preferred.

In the case where copolymerization of the crosslinking monomers (c) is desired, they are used in amounts of 0.01-20% by weight, preferably 0.02-15% by weight, particularly preferably 0.1-8% by weight.

The polymerization is carried out as precipitation polymerization in a solvent in which the monomers are soluble and the resulting polymers are of low solubility or insoluble. The solvents used according to the invention are organic solvents which have no aromatic groups and, apart from oxygen, contain no heteroatom. Suitable and preferred solvents are those selected from the group consisting of saturated hydrocarbons with 5-12 carbon atoms, dialkyl ethers with 2-12 carbon atoms, the C<sub>3</sub>-C<sub>12</sub>-ketones and C<sub>1</sub>-C<sub>22</sub>-alkyl esters of C<sub>1</sub>-C<sub>22</sub>-carboxylic acids.

Examples of suitable hydrocarbons are pentane, cyclopentane, hexane, cyclohexane, methylcyclohexane, heptane, octane or iso-octane. Examples of suitable ethers are dimethyl ether, diethyl ether, diamyl ether, tert-butyl methyl ether or dibutyl ether. Suitable ketones are dialkyl ketones such as acetone, methyl ethyl ketone, diethyl ketone or methyl amyl ketone. The reaction

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can also be carried out in alcohols such as n-butanol, 2-methyl-2-butanol, isoamyl alcohol, hexanol, cyclohexanol, octanol or decanol. Examples of suitable alkyl carboxylates are ethyl formate, methyl acetate, ethyl acetate, isopropyl acetate, isobutyl acetate, stearyl acetate, 2-ethylhexyl 2-ethylhexanoate, methyl stearate, isopropyl myristate or isopropyl palmitate. It is also possible to use mixtures of said solvents.

Preferred solvents are pentane, hexane, heptane, cyclohexane, methylcyclohexane, tert-butyl methyl ether, acetone, methyl ethyl ketone, n-butanol, methyl acetate, ethyl acetate, isopropyl acetate or isobutyl acetate. Heptane, cyclohexane or ethyl acetate are very particularly preferred. The amount of solvent is preferably chosen so that the reaction mixture can be stirred during the polymerization. The solids content of the reaction mixture is preferably in the range from 10 to 40% by weight.

It is also possible to add to the mixture small amounts of up to 10% by weight, preferably up to 4% by weight, particularly preferably up to 2% by weight, based on the monomers used, of water, methanol, ethanol, isopropanol, protective colloids or emulsifiers in order to exert a beneficial influence on the morphology of the products or the viscosity of the reaction mixture. It is also possible in this way to have a beneficial influence on other properties of the copolymers, eg. the residual contents of monomers or solvents during or after a workup step. Examples of suitable protective colloids are polyvinylpyrrolidones, partially hydrolyzed polyvinyl acetates, cellulose ethers or copolymers of N-vinylpyrrolidone and vinyl acetate. The amounts of water and/or emulsifiers present during the precipitation polymerization, if used, are only such that the mixture of all the components still appears homogeneous before the polymerization starts.

The molecular weight of the copolymers can, if desired, be reduced by adding regulators to the polymerizing mixture, for example halogen compounds such as tetrachloromethane, chloroform, bromotrichloromethane, allyl compounds such as allyl alcohol or 2,5-diphenyl-1-hexene, aldehydes, formic acid or formic esters. Polymerization regulators which contain sulfur in bound form are preferably used. Examples of compounds of this type are inorganic bisulfites, sulfites, disulfites and dithionites or organic sulfides, disulfides, polysulfides, sulfoxides, sulfones and mercapto compounds. Compounds which are particularly preferably used are mercapto alcohols, mercapto carboxylic acids and mercaptoalkanes with from two to 30 carbon atoms in the molecule, for example 2-mercaptoethanol, 3-mercaptopropanol, 3-mercapto-1,2-propanediol, 4-mercaptobutanol, cysteine, mercaptoacetic acid,



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3-mercaptopropionic acid, mercaptosuccinic acid, n-butyl mercaptan, n-hexyl mercaptan, n-dodecyl mercaptan or tert-dodecyl mercaptan. If polymerization regulators are used they are employed in amounts of 0.1-15, preferably 0.1-5, % of the weight of the 5 monomers present in the polymerization.

The monomers are subjected to free-radical polymerization, ie. compounds which form free radicals under the polymerization conditions are needed to initiate the homo- or copolymerization.

10 Initiators which form free radicals are all conventional peroxy and azo compounds, for example peroxides, hydroperoxides and peroxy esters such as hydrogen peroxide, dibenzoyl peroxide, di-tert-butyl peroxide, tert-butyl hydroperoxide, diacyl peroxides such as dilauroyl peroxide, didecanoyl peroxide and dioctanoyl peroxide or peresters such as tert-butyl peroctanoate, tert-butyl perpivalate, tert-amyl perpivalate or tert-butyl perneodecanoate, as well as azo compounds such as 2,2'-azobis(2-amidinopropane) dihydrochloride, 2,2'-azobis[2-(2-imidazolinyl)propane] dihydrochloride, 4,4'-azobis(4-cyanovaleric acid), 2,2'-azobis(2,4-dimethylvaleronitrile), 2,2'-azobisisobutyronitrile, 2,2'-azobis(2-methylbutyronitrile), dimethyl 2,2'-azobis(isobutyrate), 2,2'-azobis(4-methoxy-2,4-dimethylvaleronitrile), 1,1'-azobis(1-cyclohexanecarbonitrile), 2,2'-azobis(2,4,4-trimethylpentane) or 2-(carbamoylazo)isobutyronitrile. It is also, of 25 course, possible to use mixtures of initiators or the known redox initiators. Examples of redox initiators are combinations of at least one peroxy compound such as potassium, sodium or ammonium persulfate, sodium hypochlorite, sodium perborate, sodium percarbonate, hydrogen peroxide, tert-butyl hydroperoxide or di-tert-butyl peroxide and at least one reducing agent such as ascorbic acid, lactic acid, citric acid, sodium sulfite or sodium bisulfite, acetone sulfite, sodium dithionite, sodium N-hydroxymethylsulfinate or a tertiary amine such as dimethylaniline. The initiators are employed in conventional amounts, eg. 0.1-6% of the 35 weight of the monomers to be polymerized.

The precipitation polymerization is normally carried out under inert gas atmosphere. The polymerization can be carried out, for example, by introducing all the components present during the 40 polymerization into a polymerization vessel, starting the reaction and, if necessary, cooling the reaction mixture in order to control the temperature. However, it is also possible to start the polymerization with only a few or a portion of the components and to meter the remainder of the components in continuously or 45 batchwise, singly or together, over periods which may differ according to circumstances, depending on the progress of the polymerization. However, it is also possible for initially only

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the diluent to be present and for the monomers and the polymerization initiators to be introduced separately, batchwise or continuously.

- 5 The temperature during the polymerization is generally from 40 to 160, preferably from 50 to 120°C. It can be controlled variously during the reaction by a program. The polymerization is preferably carried out under atmospheric pressure but can also be carried out under reduced or elevated pressure. If the polymerization temperature is above the boiling point of the solvent, the polymerization is carried out in pressure-tight equipment under pressures up to 8 bar.

- The polymerization process is preferably controlled so that the resulting copolymer is in the form of a fine-particle powder. The average particle size of the polymer powder is 0.01-500, preferably 0.5-200, µm. After the polymerization, the crosslinked copolymer is separated from the other components of the reaction mixture, for example by filtration, decantation or centrifugation.
- 20 The resulting powder can be subjected, if necessary, to further suitable separating, washing, drying or milling processes.

- Those polymers obtained by the process according to the invention which are soluble in water or another suitable solvent preferably have K values (determined by the method of H. Fikentscher, Cellulose-Chemie, 13 (1932) 58-64 and 71-74 in aqueous solution at 25°C and a polymer concentration of 1% by weight) in the range from 10 to 200.

- 30 After the reaction, the polymers can be converted in the same medium, or after isolation in another medium, by a suitable reagent to a quaternized form. Suitable for the quaternization are, for example, alkyl halides having 1-18 carbon atoms in the molecule; eg. methyl chloride, ethyl chloride, propyl chloride, hexyl chloride, dodecyl chloride or lauryl chloride, as well as benzyl halides such as benzyl chloride. The corresponding iodine or bromine compounds are also, of course, suitable. Further suitable quaternizing agents are dialkyl sulfates, in particular dimethyl sulfate and diethyl sulfate. In some cases it is also sufficient to convert the polymers into the salt form by treatment with an acid. The quaternization can take place completely or partially.

- The polymers obtained according to the invention can be used, for example, for binding bile acids in the blood to reduce the cholesterol level or for selective removal of heavy metal ions from solutions, and as auxiliaries in cosmetic formulations, for

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example to adjust the rheological behavior. The polymers according to the invention can also be used as detergent additive to inhibit transfer of dyes during the washing process.

## 5 Examples

### Example 1

400 g of ethyl acetate, 100 g of N-vinylimidazole, 10 g of divinylethyleneurea and 1 g of tert-butyl perpivalate were stirred at 72°C in a 2000 ml flask which was equipped with a stirrer, reflux condenser, thermometer and an apparatus for working under protective gas for 2 hours. The resulting product was filtered off on a suction funnel, washed with 100 g of ethyl acetate and dried in a vacuum oven at 50°C for 8 hours. 111 g of a white powder with an apparent density of 67 g/100 ml and an average particle size of 124 µm were obtained.

### Example 2

600 g of cyclohexane, 100 g of N-vinylimidazole, 10 g of divinylethyleneurea and 1 g of azobisisobutyronitrile were stirred at 77°C in a 2000 ml flask which was equipped with a stirrer, reflux condenser, thermometer and an apparatus for working under protective gas for 4.5 hours. The resulting product was filtered off on a suction funnel, washed with 200 g of cyclohexane and dried in a vacuum oven at 50°C for 8 hours. 109 g of a white powder with an apparent density of 6 g/100 ml and an average particle size of 30 µm were obtained.

### Example 3

400 g of methyl ethyl ketone, 100 g of N-vinylimidazole, 10 g of allyl methacrylate and 1 g of 2,2'-azobis(2-methylisobutyronitrile) were stirred at 74°C in a 2000 ml flask which was equipped with a stirrer, reflux condenser, thermometer and an apparatus for working under protective gas for 2.5 hours. The resulting product was filtered off on a suction funnel, washed with 100 g of methyl ethyl ketone and dried in a vacuum oven at 50°C for 8 hours. 106 g of a white powder with an apparent density of 10 g/100 ml and an average particle size of 11 µm were obtained.

### Example 4

400 g of cyclohexane, 50 g of N-vinylimidazole, 50 g of N-vinylpyrrolidone and 2 g of divinylethyleneurea were heated to 77°C in a 2000 ml flask which was equipped with a stirrer, reflux

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condenser, thermometer and an apparatus for working under protective gas. As soon as this temperature was reached, 0.5 g of dimethyl 2,2'-azobisisobutyrate was added dropwise over the course of 2 hours. The mixture was then stirred at this temperature for a  
5 further 4 hours. The resulting product was filtered off on a suction funnel, washed with 200 g of cyclohexane and dried in a vacuum oven at 50°C for 8 hours. 101 g of a white powder with an apparent density of 32 g/100 ml and an average particle size of 12 µm were obtained.

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## Example 5

400 g of cyclohexane were heated to 77°C in a 1000 ml flask which was equipped with a stirrer, reflux condenser, thermometer and an  
15 apparatus for working under protective gas. As soon as this temperature was reached, 70 g of 1-methyl-2-vinylimidazole and 0.7 g of mercaptoethanol were added dropwise over the course of 1 hour and, in parallel to this, 0.4 g of azobisisobutyronitrile in 20 ml of cyclohexane was added dropwise over the course of  
20 2 hours. The mixture was then stirred at this temperature for a further 4 hours. The resulting product was filtered off on a suction funnel, washed with 200 g of cyclohexane and dried in a vacuum oven at 50°C for 8 hours. 68 g of a white powder were obtained. A solution of the polymer in water was clear and color-  
25 less and had a K value of 26.2 (determined by the method of H. Fikentscher, Cellulose-Chemie 13 (1932) 58-64 and 71-74 at 25°C and a polymer concentration of 1% by weight).

## Example 6

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400 g of ethyl acetate, 100 g of N-vinylimidazole and 17.3 g of stearyl acrylate were heated to 70°C in a 2000 ml flask which was equipped with a stirrer, reflux condenser, thermometer and an  
35 apparatus for working under protective gas. As soon as this temperature was reached, 1 g of tert-butyl perpivalate in 20 ml of cyclohexane was added dropwise over the course of 2 hours. The mixture was then stirred at this temperature for a further 4 hours. The resulting product was filtered off on a suction funnel, washed with 200 g of cyclohexane and dried in a vacuum  
40 oven at 50°C for 8 hours. 116 g of a white powder were obtained. A solution of the polymer in ethanol had a K value of 31 (determined by the method of H. Fikentscher, Cellulose-Chemie 13 (1932) 58-64 and 71-74 at 25°C and a polymer concentration of 1% by weight).

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## Example 7

15 g of the polymer powder from Example 1 were dispersed in 200 g of ethanol and, at 40°C, 30 g of dimethyl sulfate were added over 5 the course of 30 minutes. The mixture was subsequently heated to 60°C and stirred at this temperature for a further 3 hours. The product was then filtered off on a suction funnel, washed with 100 ml of water, stirred twice in 400 ml of a 10% strength aqueous NaCl solution for 15 minutes, again filtered off with 10 suction, washed with twice 100 ml of water and dried in a vacuum oven at 50°C for 8 hours. 21 g of a polymer powder with an exchange capacity of 5.8 meq/g were obtained.

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